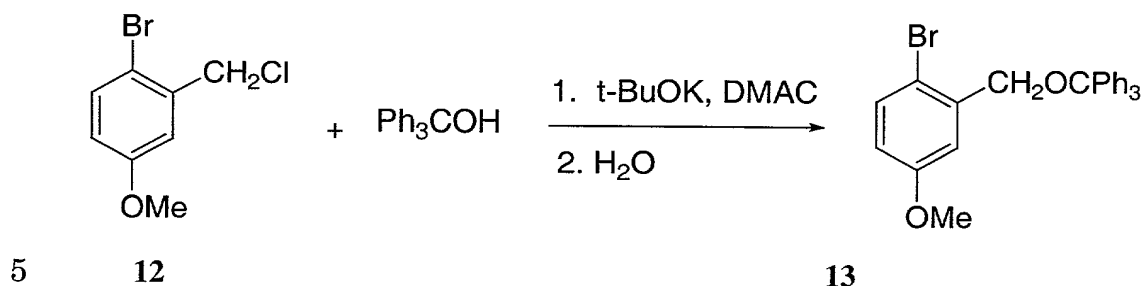


### EXAMPLE 7

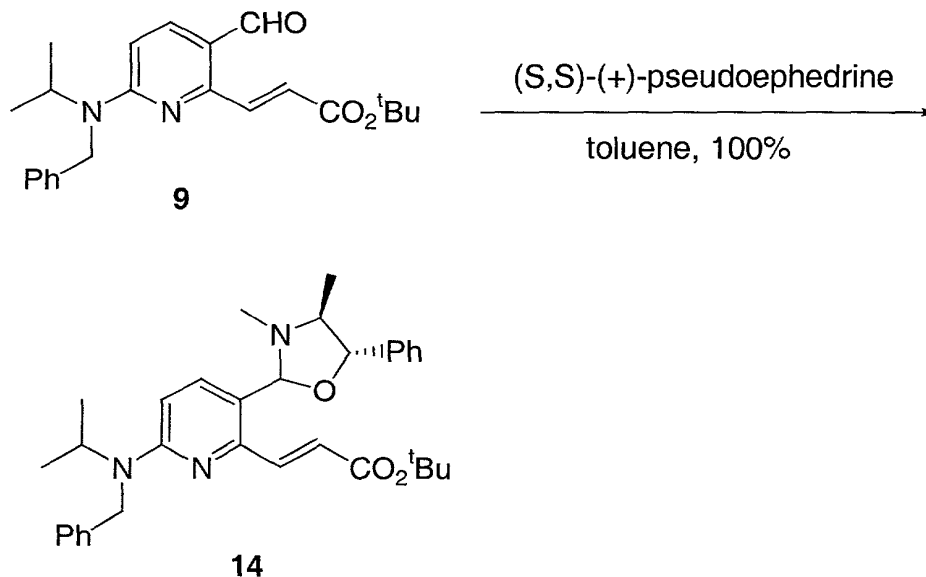
2-Bromo-5-methoxybenzyl trityl ether:



Under nitrogen, dimethylacetamide, (DMAC, 3.14L), Ph<sub>3</sub>COH (573g, 2.2mol) and *tert*-BuOK (236g, 2.1mol) are sequentially added to a three-necked 12L flask, and then 2-bromo-5-methoxybenzyl chloride **12** (470g, 2.0mol) in DMAC (0.66L) is added over an hour. The reaction mixture is stirred at room temperature for another hour. About 1.26L of water is slowly added to the reaction mixture over an hour to crystallize the product. The slurry is stirred at room temperature for another hour and then filtered. The wet cake is washed with about 3L of 80:20 DMAC:H<sub>2</sub>O and water. The cake is dried by vacuum suction under nitrogen for 12 hours to give the compound **13** (800g, 99.5W%, 99.7A%) as a bright white crystalline solid. The use of 80:20 DMAC:H<sub>2</sub>O is recommended for washing to remove by-products formed in the reaction. The additional wash with water can remove inorganic substances, such as KCl. By-products, dibenzylether and stilbene, formed in the reaction can also be removed by crystallization.

**HPLC conditions:** Zorbax RX-C8, 4.6 x 150; MeCN/H<sub>2</sub>O at 1.5mL/min; UV detector at 220nm; Retention times (min): Ph<sub>3</sub>COH (8.5), dibenzyl ether (12.5), stilbene (13.6), and trityl ether **13** (16.4).

## EXAMPLE 8

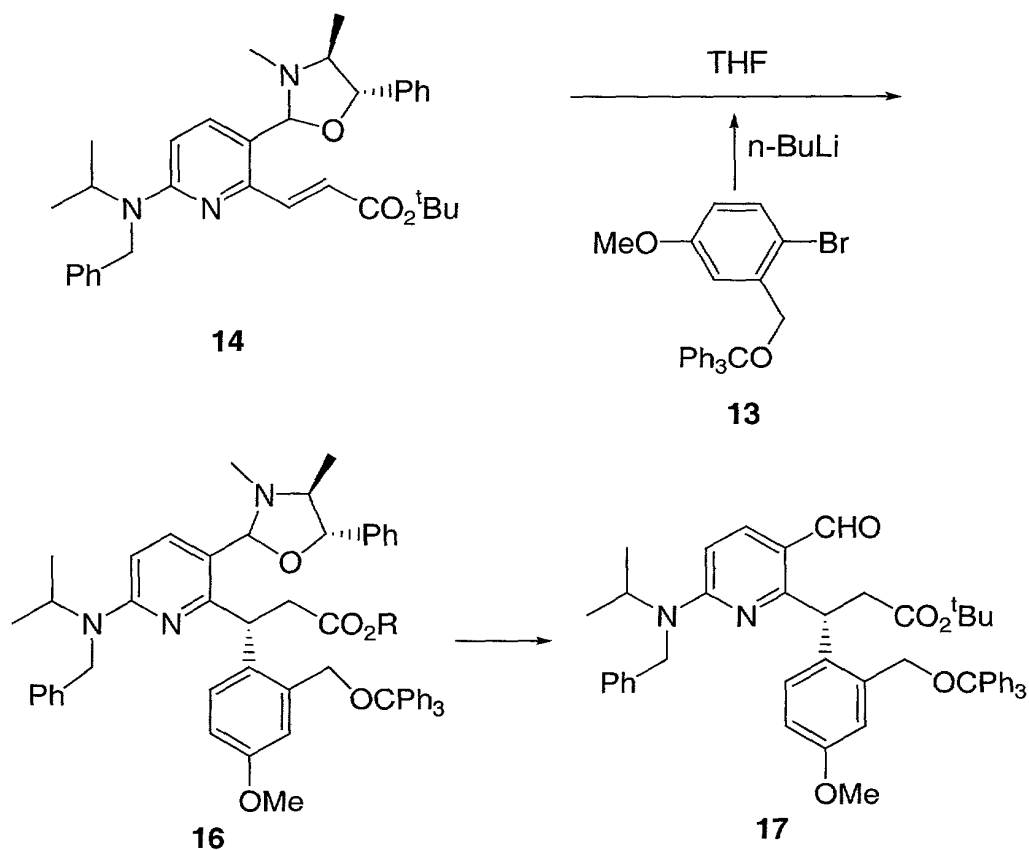
N,O-Acetal Formation:

5                    A 3L three-neck round bottom flask equipped with a mechanical stirrer, nitrogen line, Dean-Stark trap with condenser and temperature probe is charged with toluene (0.93 L, KF=52 $\mu$ g/mL) and the Heck product **9** (185.8g). To the solution, (S,S)-pseudoephedrine (104.1g) and camphorsulfonic acid (csa, 2.7g) are added. The reaction mixture is then refluxed vigorously until **9** is completely

10 consumed. Upon cooling the mixture to about room temperature, Florisil (93 g) is added and the slurry is stirred for about 30 minutes. The Florisil is then filtered off and washed with toluene. The filtrate and wash are combined and washed with water. The organic layer is concentrated to about 1.7L. The solution is flushed with toluene

15 until the KF is 250  $\mu$ g/mL.

## EXAMPLE 9

Conjugate Addition:

- 5 A 12L three-neck round bottom flask equipped with a mechanical stirrer, nitrogen line and temperature probe is charged with aryl bromide **13**. The flask is then purged with nitrogen. Degassed toluene (2.1 L, KF=84 $\mu$ g/mL) and THF (2.1 L, KF=278 $\mu$ g/mL) are then charged, and the flask is purged with nitrogen. The solution is cooled to about -70°C and 1.6M *n*BuLi (537 mL) is added by using a gas tight syringe over 25 minutes. The solution is aged for 15 minutes and then checked by HPLC for residual ArBr. When ArBr is completely consumed, a solution of **14** in about 1.7L toluene is added to the reaction mixture via canula over 20 minutes. The reaction mixture is aged for about 25 minutes, and then warmed to about -50°C and quenched by the addition of HOAc (179mL). The mixture is again allowed to warm to about 0°C. Aqueous citric acid (333 g citric acid + 930mL water) is added, and the
- 10
- 15